june 1959

nlgi spokesman

journal of the national lubricating grease institute

Yield Points of Lubricating Greases

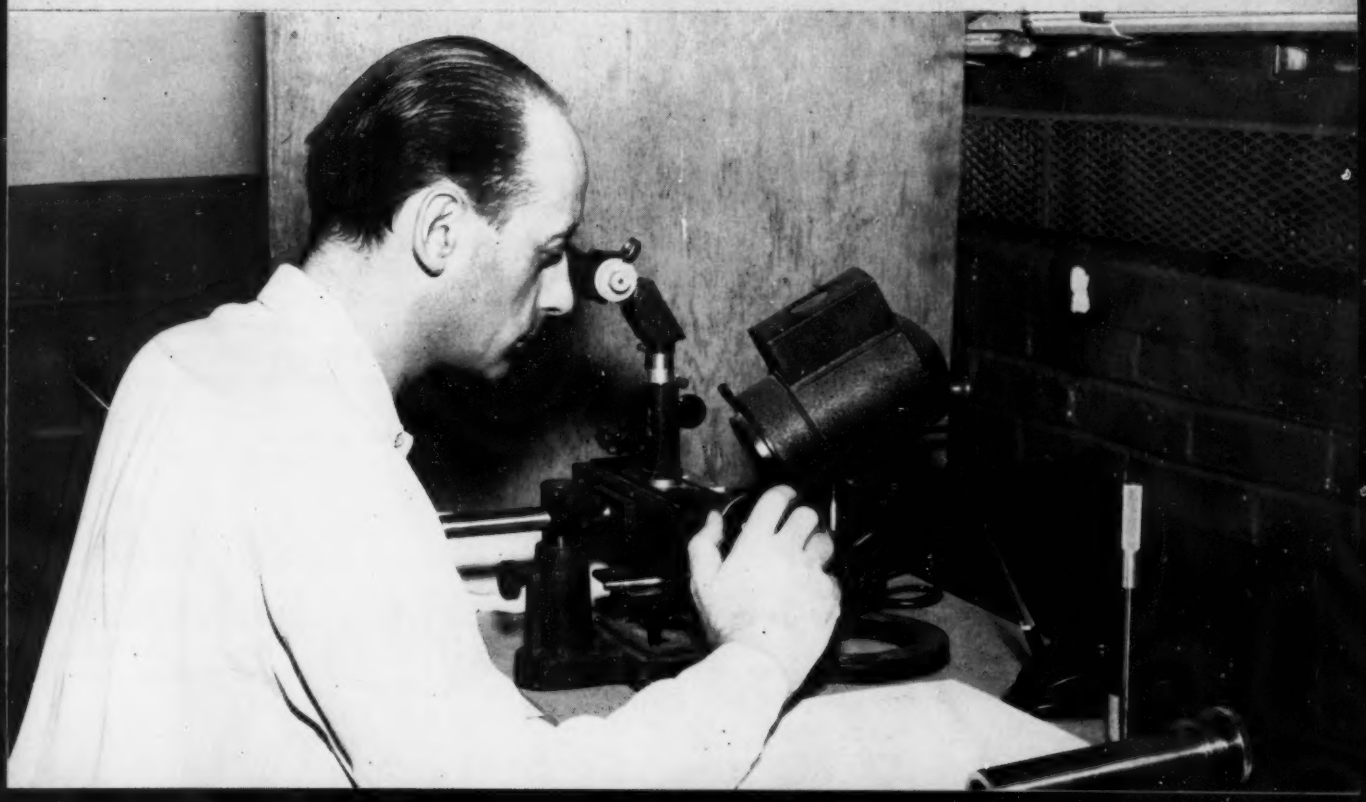
By D. W. CRIDDLE and J. L. DREHER

A Reproducible Rust Test for Greases

By K. R. FISCH, J. MESSINA and H. GISSER

How to Get the Motorist to Lubricate Every 1,000 Miles

By M. M. BORSKI



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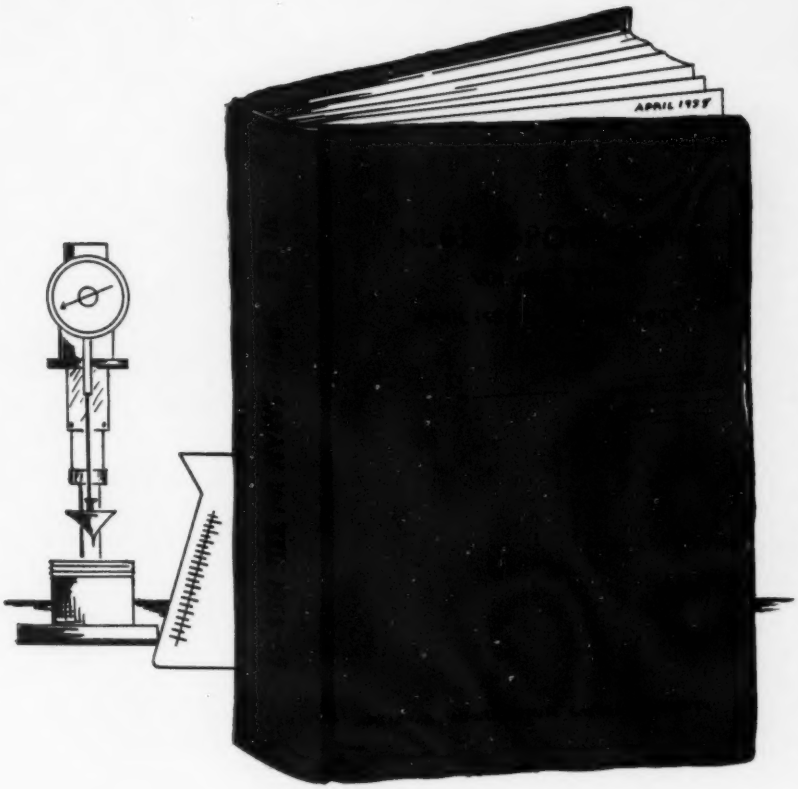

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NLGI PRESIDENT'S PAGE

By F. E. ROSENSTIEHL, *President*

Present Status of Automotive Gear Lubricants

There appears to be a good deal of confusion regarding the present status of automotive gear lubricants. Some time ago it was recognized that the increased horsepower and torque characteristics of the new higher output engines, plus decreased rear axle gear ratios, were placing rather severe demands on current lubricants. In particular, the lubricants left something to be desired in meeting passenger car needs and giving satisfactory protection to hypoid gears from a standpoint of anti-score and anti-scuff, and in some instances oxidation and corrosion resistance. In an attempt to meet this problem new test methods to evaluate and define anti-score and anti-wear properties were developed by the Coordinating Research Council. As a result a large number of improved gear lubricants have been developed and are now on the market and generally available at service stations.

Extensive work has been done in representative field test equipment to determine the effect of mileage change intervals on these newer lubricants. The tests indicate that under high output performance even some of the higher load carrying products showed appreciable deterioration at mileages as low as 10,000 miles. The information derived from these tests provide additional justification for adopting a generally lower mileage change interval in both passenger and commercial equipment. Despite this many 1959 model cars do not have the drain plugs reinstated, thus preventing drain and refili.

As the new lubricants became more widely distributed the API Lubrication Committee developed a new service definition by which the automotive manufacturers could recommend these lubricants, namely "API Service GL-4." This API designation is listed in the 1959 SAE Handbook. It is the responsibility of the lubricant manufacturers to determine whether or not their product is suitable for this type service. While this definition has been used by several car manufacturers in their car owners instruction manuals, unfortunately this has not been adopted by all. Further, many car manufacturers continue to buy lubricants of their particular specifications for factory fill.

More recently certain of the car manufacturers have requested a more definitive identification of lubricants suitable for API Service GL-4 and it is hoped that as both industries develop more experience with the newer gear lubricants, some type of correlating interpretation can be worked out between the Automotive and Petroleum industries.

Recently the government issued new specification, MIL-L-002105A (ORD), which calls for gear lubricants of improved performance characteristics. This is a definite specification and not a service designation, and it should be understood that neither the API GL-4 designation nor the new military specification requires that one be a part of the other, though such may well be the case with many of the newer gear lubricants.

Many are erroneously of the opinion that products which meet the new government specification or which are satisfactory for service under the API GL-4 designation are automatically suitable for limited-slip differentials. This, unfortunately, is not necessarily the case, since most car manufacturers require products of special additional characteristics, particularly from an antifriction standpoint for use in this type differential.

It is hoped that the confusion which now exists will be eliminated in the near future.

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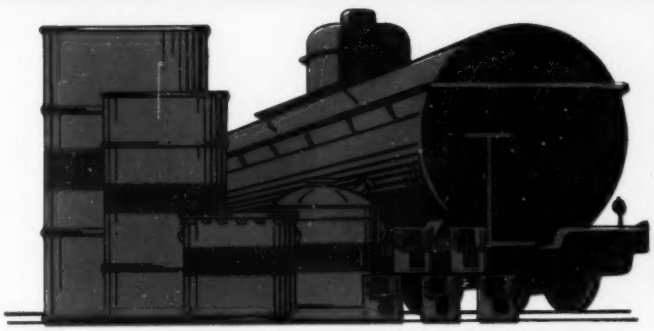

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News About NLGI

A. S. Randak

The NLGI Board of Directors passed a unanimous resolution of sympathy and condolence to the family of A. S. Randak, former member of the Board and long time Company Representative to NLGI from the Sinclair Refining company. Mr. Randak died Monday, May 4, shortly before he was to assume new duties with Sinclair as Eastern assistant division manager. Prior to the announcement of this promotion, Mr. Randak had been manager of lubricating sales, and it was the change of responsibilities that had necessitated his resignation from NLGI's governing body.

Second NLGI Survey Released to Members

The second annual survey on the production of lubricating greases and fluid gear lubricants, for the calendar year of 1958, has been compiled and released to the membership. Distribution was completed last month.

The summary of totals gave a comparison of figures for 1957, when the first survey was taken, and also gives totals for Canada . . . 1958 was the first year for the NLGI members in the Dominion. Statistics were gathered by the management services division of Ernst & Ernst, nation-wide certified public accountants and delivered to the NLGI national office for mailing. The committee for the 1959 survey is currently studying 1958 results for modification and improvement in the questionnaire, and will announce the changes later in the year.

Members of NLGI receive survey results as a membership service. Non-member firms may obtain copies for $10 by writing the national office.

Early Bird Reception

Because of an anticipated early registration on the day before the October 26-28 Annual Meeting at the Roosevelt hotel in New Orleans, a special "Mr. and Mrs. Early Bird Reception" will be held for members and wives on Sunday, October 25 . . . this will be the first function of the 27th Annual Meeting.

The decision to hold this new event was made at the Board of Directors meeting in Chandler, Arizona, on May 7. Further announcements concerning the program, the largest in NLGI history, will be made in future issues of the journal.

Technical Committee Column

CHAIRMAN T. G. ROEHNER

Socony Mobil Oil Company, Inc.

Manager

Technical Service Laboratory

In accordance with approvals received at the 1958 Annual Meeting, the scope of the committees reporting to the NLGI Technical Committee will be expanded.

Organization has been started on a new Technical Committee on Gear Lubricants. The first chairman is Dr. R. K. Smith of E. F. Houghton and company. It will focus its attention primarily on industrial gear problems because SAE already has a strong program on automotive gear lubricants.

A Technical Committee on Manufacturing Operations is being or-

ganized, with Mr. J. J. Dickason of Jesco Lubricating company as its first chairman. Top priority has been assigned to this new committee because they have been requested to conduct a half-day session of manufacturing problems at the next Annual Meeting in New Orleans. In other words, this new group will be following the pattern adopted by the new Technical Committee on Fundamental Research who kicked off their first annual meeting with a symposium on rheology of lubricating greases last October in Chicago.

The responsibilities of the Technical Committee on Fundamental Research has been expanded to include those of the Technical Committee on the NLGI Research Fellowship. Dr. J. J. Kolfenbach, of Esso Research and Engineering company, resigned as chairman of this committee last year, and Mr. L. C. Brunstrum of Standard Oil company (Indiana), formerly NLGI Technical Committee vice-chairman, is now chairman, with Mr. E. L. Armstrong of Socony Mobil Oil company, Inc., as the new vice-chairman.

Another new committee is being established on consistency problems, with Mr. M. Ehrlich of American Lubricants, Inc. as the first chairman. They will take over the responsibilities formerly handled by the Technical Committee on the NLGI Classification. Their main assignment will be to keep NLGI up to date relative to the activities of other organizations, but particularly ASTM Technical Committee "G," on projects involving consistency of lubricating greases.

The Chairmen of the above new committees would appreciate information concerning candidates for membership in their respective groups and also recommendations regarding the scope which their committees should cover in order to be of the most service to NLGI.

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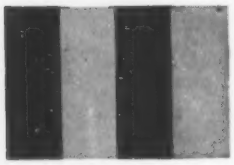

Future Meetings

MAY, 1959

31-June 6 Fifth World Petroleum Congress and Exposition. The Coliseum, New York City.

JUNE, 1959

1-5 Fifth World Petroleum Congress Exposition, The Coliseum, New York City.

8-9 Petroleum Packaging Committee Meeting, Cleveland Ohio.

14-19 SAE Summer Meeting, Chalfonte-Haddon Hall, Atlantic City, N. J.

21-26 ASTM Annual Meeting Chalfonte-Haddon Hall, Atlantic City, N. J.

AUGUST, 1959

16-21 1959, National Congress of Petroleum Retailers Inc., 13th Annual Session, Jung Hotel, New Orleans, La.

SEPTEMBER, 1959

16-17 Ohio Petroleum Marketers Association, Fall Conference and Golf Tournament, Sheraton-Cleveland Hotel and Lakewood Country Club, Cleveland, Ohio.

27-29 IOCA 12th Annual Meeting, Pick-Congress Hotel, Chicago, Ill.

OCTOBER, 1959

26-28 NLGI Annual Meeting, Roosevelt Hotel, New Orleans, La.

11-15 ASTM Committee D-2 Meeting, Sheraton - Palace Hotel, San Francisco.

12-13 Petroleum Packaging Committee Meeting, Port Arthur, Texas

19-21 ASLE and ASME Joint Lubrication Conference, Sheraton-McAlpine Hotel, New York City.

28-30 Society of Automotive Engineers, National Fuels and Lubricants, La Salle Hotel, Chicago.

NOVEMBER, 1959

9-11 API, 39th Annual Meeting, Conrad Hilton, Palmer House and Congress Hotels, Chicago

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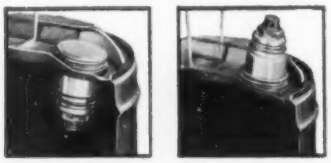

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Volume XXIII JUNE, 1959 Number 3

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THE COVER

K. R. FISCH, chemist for the U. S. Army Ordnance Corps, Frankford Arsenal, Philadelphia, is shown measuring the grease film thickness of a test specimen employing the use of a traveling microscope. Mr. Fisch in collaboration with Mr. Henry Gisser and Mr. Joseph Messina, both of whom are also chemists with the U. S. Army Ordnance Corps, have co-authored the paper "A Reproducible Rust Test for Greases" which begins on page 102 and deals with the development of a means for producing a test specimen with reproducible grease film thickness, as part of a grease-rust test.

The NLGI SPOKESMAN is indexed by Industrial Arts Index and Chemical Abstracts. Microfilm copies are available through University Microfilm, Ann Arbor, Mich. The NLGI assumes no responsibility for the statements and opinions advanced by contributors to its publications. Views expressed in the editorials are those of the editors and do not necessarily represent the official position of the NLGI.

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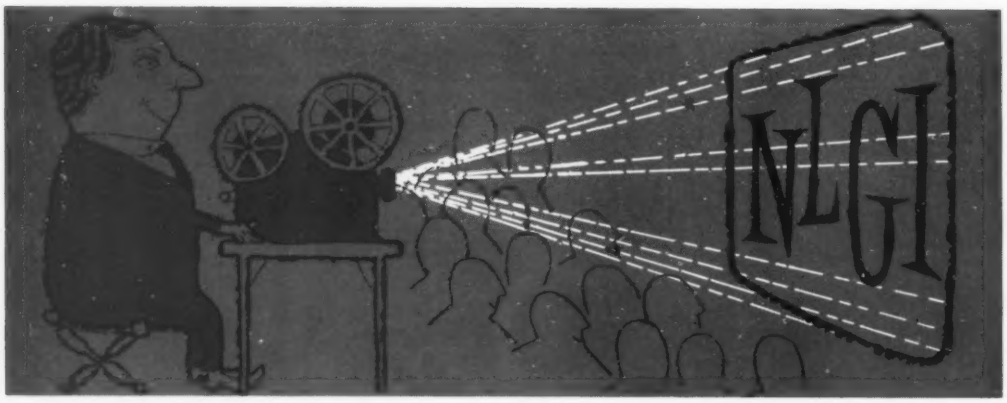
nlgi film price cut

Members and friends of the National Lubricating Grease Institute are now in a position to employ a vital selling and public relations tool at greatly reduced cost . . . as announced before, (NLGI SPOKESMAN, April) the cost of "Grease—the Magic Film" has been reduced.



The 25-minute movie was produced and subsidized by the Institute to tell the story of lubricating grease. With sales now double the initial estimate, the board of directors reduced the cost of first prints from $600 to $300, while second and all subsequent prints were priced from $400 to $200. The third price category was discontinued (non-members still add $100 to each category).

"Grease—the Magic Film" may be purchased through the national office of NLGI and delivery can usually be made within two weeks of receipt of order. A preview copy of the picture may also be obtained from NLGI upon request, along with scripts if foreign voice narration is anticipated.

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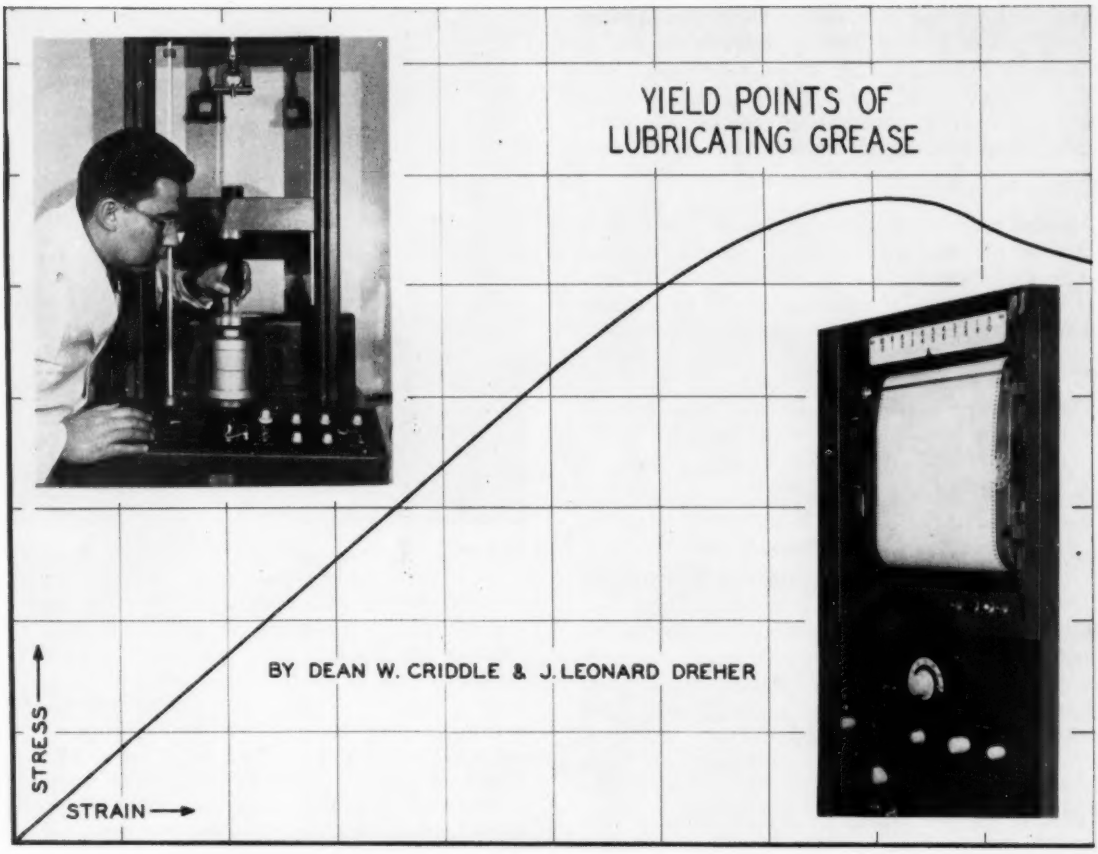

Presented at the NLGI 26th annual meeting in Chicago, October 1958

Fundamental Research – 3

Brief Abstract Indicating Objective and Scope

This paper points out that the yield point of a grease depends upon three variables: the rate of deformation, the type of deformation (compressive or shear), and the work-history of the grease. Yield points were obtained by five different methods and are compared and discussed in terms of the above three variables.

Yield point, or yield value, is a frequently used term in the lubricating grease industry. Despite its common usage, however, it is usually not clearly defined. A number of methods have been used to measure it, with widely varying results. In this paper we compare the results obtained by several methods, comment on their significance, and discuss the reasons for differences in the results. The yield points are reported as yield stresses, although both a yield strain and a yield stress characterize the yield point.

Methods

Cone Penetration

The penetration of a cone has been used to determine the "yield point" of a grease.[1,2] This method should be used only on greases hard enough to bring a falling cone to rest. The grease structure then supports the weight of the cone and assembly. From the weight and the cross-section of the cone at the grease surface, a yield point, or more specifically, a yield stress, is calculated. A buoyancy correction is made assuming the cone displaces a fluid having the density of the grease.

We used the ASTM penetration as one measure of yield point. The relationship between the ASTM penetration and yield point is shown in Figure 1.

This method is difficult to interpret because it involves a combination of shear and compressive deformation, and it uses varying amounts and rates of shear.

Capillary Viscometer

Another method of obtaining "yield points" is to determine the minimum pressure required to cause flow in a capillary.[3] We used a modified ASTM Pressure Viscometer, and the data reported were obtained at a minimum discernible flow rate of 0.0020 cc/sec.

New Flow Equation

The following equation was recently developed for calculating the flow of greases in capillaries.[4]

$$\frac{v}{t} = \left[\frac{P\pi r^4}{8LN_o}\right] \left[\frac{1}{1 + 2.5V + 14.0V^2}\right] [C]$$

$\frac{v}{t}$ is the flow rate, P is pressure, r and L are the capillary radius and length, respectively, N_o is the oil viscosity, and V is the volume per cent solids. The term C is a function of the applied pressure and a "yield point" of the grease. With the above equation, yield points for flow were calculated.

Stress-Strain Curves

"Yield points" for greases are readily obtained from stress-strain (force-deformation) curves. A convenient and precise apparatus for obtaining these curves is shown in Figure 2. Through gears, the upper platen is driven at a controllable rate towards or away from the stationary lower platen. The rate can be varied from 0.005 to 50 cm/min. The force required to shear or compress samples of grease placed between the two platens is detected by a strain gage connected to the lower platen and is automatically recorded. The instrument is reliable to ± 2 per cent over a range of stresses from one-quarter gram to a million grams.

Stress-strain curves were obtained for deformations in shear and in compression. For studies of yield points

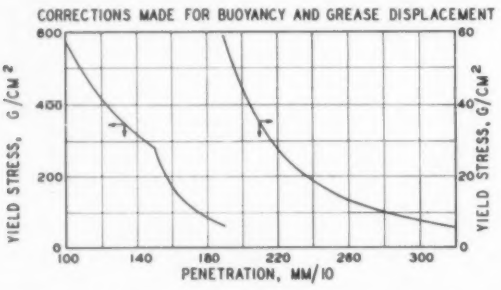

FIGURE 1, yield stress from ASTM cone penetration data

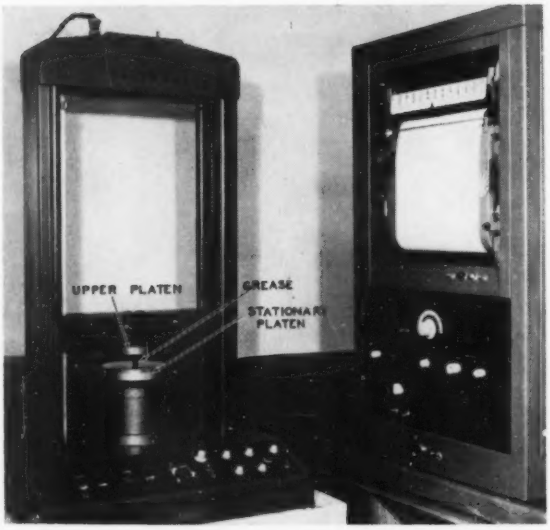

FIGURE 2, apparatus used to determine stress-strain curves

in shear, samples were mounted as shown in Figure 3. The annular space between two glass cylinders 1.77 cm OD and 2.61 cm ID and 10 cm long was filled with grease. Before the samples were prepared, the greases were worked 60 strokes in the ASTM worker. After preparation, they were allowed to set for ten to fourteen days before testing. Tests were run at 25 ± 1°C. The aged samples were placed between the platens, and the inner cylinder was forced to move with respect to the outer at a predetermined rate. The grease sheared at the inner cylinder. The total stress divided by the area of the inner cylinder in contact with the grease was taken as the shear stress. The standard deviation of repeat runs was 15 per cent.

A typical stress-strain curve is shown in Figure 4. At low stresses and strains, the grease is elastic and the curve is essentially a straight line. As the stress is increased (at a constant rate of shear) the elastic limit of the grease is exceeded and the curve deviates from

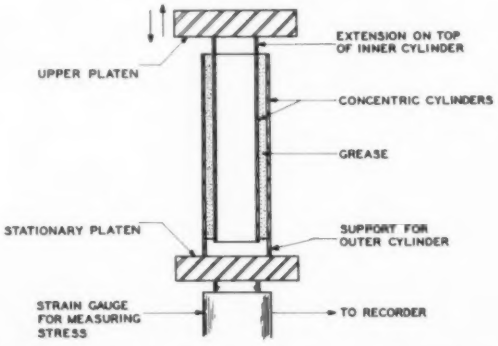

FIGURE 3, apparatus and sample used to determine yield points in shear

a straight line. The point of deviation is defined by rheologists as the yield point.[5] As the stress is further increased, a maximum is reached. This point is commonly called the ultimate yield point.[6] The stress-strain curve for a lubricating grease is similar to that obtained by Bridgman for metals.[7] In the case of metals, the stresses are greater by a factor of at least a million.

Studies of yield points in compression were made on unsupported cylinders of grease, which were prepared by extruding grease from cork borers. The samples were worked and aged as in the shear experiments. The lengths of the cylinders were at least twice the diameters in order to minimize end effects.[8] After prepared samples were aged from ten to fourteen days, stress-strain curves in compression were obtained at $25 \pm 1°C$. A typical stress-strain curve in compression is similar to that in shear (Figure 4) except that greater stresses are necessary to produce a given strain.

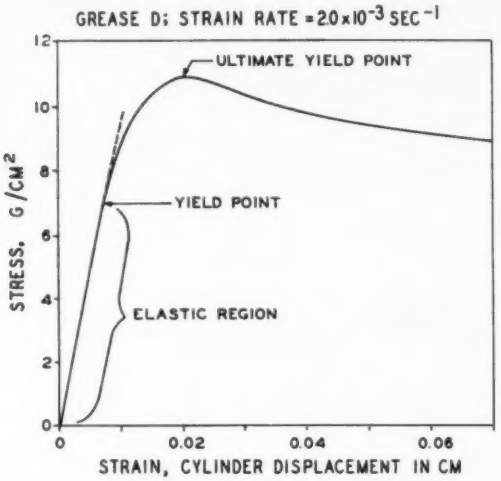

FIGURE 4, typical shear stress-strain curve for grease

Buckingham Equation

A "yield point" can be calculated by applying the Buckingham equation to flow data.[9,10] This equation is based on an ideal Bingham solid which starts to flow when the shear stress exceeds the Bingham yield point. We applied the Buckingham equation to capillary flow data and solved for the "Bingham yield point." For a narrow range of shear rates, this two-parameter equation can be made to fit experimental data in terms of a Bingham viscosity and the Bingham yield point. However, the yield points calculated by this method varied threefold, depending on whether the high or low shear rate data were fitted by the equation. Because the "Bingham yield point" depends upon the range of shear rates chosen to obtain a best fit of the equation with experimental data, we do not consider this yield point to be fundamentally significant. Therefore, we are omitting Bingham yield points in this comparison.

TABLE I

Composition and Properties of Test Greases*

Grease Sample	*Thickener Type*	*Wt %*	*ASTM Penetration Unworked*	*Worked*
A	Sodium Oronite GA-10†	5	336	354
B	Sodium Oronite GA-10	7	265	305
C	Sodium Oronite GA-10	9	225	251
D	Sodium Oronite GA-10	11	197	216
E	Sodium Oronite GA-10	13	174	189
F	Sodium Oronite GA-10	15	164	167
G	Lithium Stearate	14	152	216

* Base oil had a viscosity of 2.30 poises at 25°C.
† Sodium salt of methyl N-octadecylterephthalamate.

Data and Discussion

Seven greases, whose composition and properties are given in Table I, were used to study yield points. The values calculated from data obtained by the different methods are given in Table II.

The rate of deformation affects the results, as shown by the stress-strain data in Figures 5 and 6. In both shear and compression, the yield stress descreases as the deformation rate decreases. This indicates that there is no single "yield stress." It is probable that at very low pressures lubricating greases creep as do many solid materials, including metals. The detection of the creep depends upon the sensitivity of the instruments used to measure flow and pressure.

The above data show that for a given grease, the yield stress depends upon at least three factors: the type of deformation, the rate of deformation, and the previous work history of the grease. These three fac-

TABLE II

Comparison of Yield Stresses Determined By Different Methods

Stresses given in g/cm²

Grease Sample	*Calculated From ASTM Penetration Unworked*	*Worked*	*Capillary Viscom.**	*New Flow Equa.*	*Stress-Strain Shear†*	*Curve Compress‡*
A	4	**	4	2.5	1	**
B	12	5	12	6	3	**
C	25	15	16	9	5	19
D	45	28	27	22	10	38
E	98	57	38	25	16	59
F	140	125	54	30	28	90
G	240	28	—	—	9	50

*Capillary dimensions: r = 0.0215 cm; L = 1.52 cm.
†Ultimate yield point determined at a deformation rate of 2.0×10^{-3} sec^{-1}.
‡Ultimate yield point determined at a deformation rate of 3.3×10^{-4} sec^{-1}.
**Grease too soft.

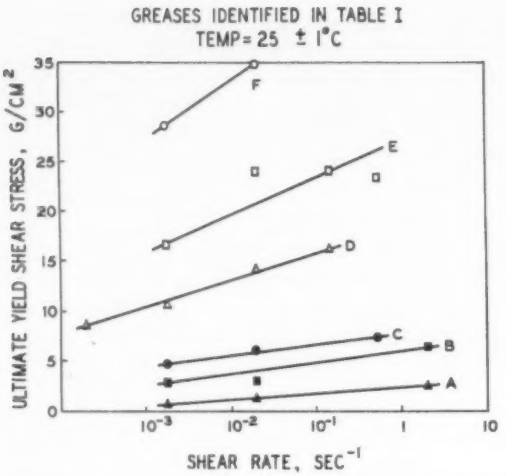

FIGURE 5, effect of shear rate on ultimate yield stress in shear

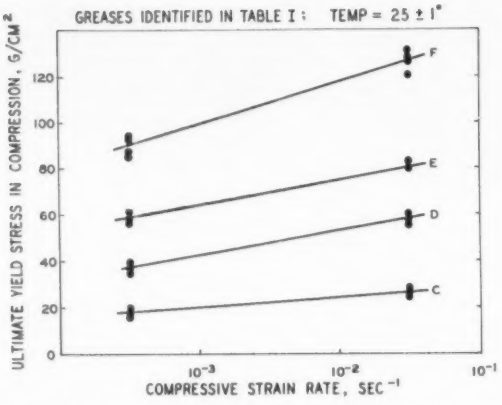

FIGURE 6, effect of shear rate on ultimate yield stress in compression

tors are discussed below:

1. *Type of Deformation*—A comparison of the data obtained in "shear" and "compression" by the stress-strain curve method shows that the type of deformation is the *principal* cause for differing results. Yield points measued under compressive conditions averaged four times greater than those measured under shear conditions. According to theory,[8] the compressive yield point of a perfect solid is greater than the shear yield point by a factor of 3.5.

The high values obtained by the penetration method indicate that this method measured compressive yield points primarily. The capillary viscometer and flow equation methods appear to give yield points in shear. With more sensitive methods of detecting flow, the viscometer method undoubtedly would give lower values, more in line with those obtained for shear by the stress-strain curve method.

2. *Rate of Deformation*—The yield stress both in shear and in compression increased an average of about 40 per cent when the strain rate increased 100-fold. Methods involving widely different rates of strain should differ because of this effect. This is believed to be a secondary but real effect, because from an extrapolation of our data strain rates varying 10,000-fold are required to increase the yield point as much as 100 per cent.

If greases behave as a model of one spring and one dashpot in parallel (Kelvin Model), we would expect the yield point to increase linearly with strain rate.[11] However, we find that yield stress increases with the logarithm of the strain rate. Greases are more complex than a simple Kelvin model.

3. *Work-History*—The difference between the yield point of unworked and worked grease samples explains

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to some extent the difference between the results of other methods. Working usually softens a grease; consequently, as shown in Table II, the yield point of a worked grease is usually less than that of the unworked grease. The generally accepted explanation is that the working process destroys more thickener junctions than it creates. At the time of testing by the various methods, the samples have not necessarily received the same amount of working or aging.

Summary

The yield points of lubricating greases were determined by five different methods. It was found that:

1. The "yield point" is dependent upon the test method.
2. Compressive yield points should be and are higher than shear yield points. This explains to a large extent the dependency of yield point upon test method.
3. The "yield point" increases as the deformation rate increases both for shear and compressive stresses.

We conclude that greases have many yield points, depending upon the type and rate of deformation. Therefore, the conditions under which yield points are obtained should be reported with the yield values.

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Acknowledgement

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A Reproducible Rust Test For Greases

By: K. R. Fisch
J. Messina
H. Gisser
U. S. Army Ordnance Corps

STUDIES ON INSTRUMENT greases conducted in the tropics during and shortly after the war years indicated a need for improvement of their rust preventive properties (1). Neither the fatty acid metal soaps nor the non-soap gelling agents provide adequate rust protection per se. As a result, rust inhibitors for greases found increased use. The development of a technique for the evaluation of the rust protective properties of greases became therefore mandatory. The CRC recently completed the development of such a test method (L41-957) (2). This test consists of lubrication and storage of bearings in a tightly closed jar containing a little water at 77°F. for fourteen days.

The CRC procedure is a qualitative "go-no go" method which differentiates between inhibited and uninhibited greases but is not too satisfactory in making a quantitative distinction between greases whose rust preventive properties are not widely separated. There is still a need for a method that will enable distinguishing the difference in rust-preventive properties of greases made from good, indifferent, and poor rust inhibitors. Such a method is needed not only for use in specifications, but also in research on rust-preventive greases.

One of the major factors associated with reproducibility, or the lack thereof, in rust-preventive tests on grease is the control of film uniformity. Another important factor is the control of the environment in which the rust-preventive test is run, e.g. the humidity cabinet. In this work, we are primarily concerned with the development of a means for producing a test specimen with reproducible grease film thickness as part of a grease-rust test.

The most common method employed in producing films of controlled thickness is the Doctor Blade (3). Devices utilizing this principle, such as the Automatic Doctor Blade (ASTM method D823-45T), the Film-O-Graph, and many others are described in detail in Gardner and Sward (4). These methods deal with

Editor's Note—The NLGI SPOKESMAN *has always maintained a policy of welcoming technical and marketing papers other than those presented at the Annual Meetings . . . features such as the above article by Fisch, Messina and Gisser may be offered the Institute, subject to the conditions of review that all papers receive. The procurement committee for the journal invites authors to present work on lubricating greases and fluid gear lubricants, for future inclusion in the publication.*

paint films applied to flat surfaces and are reasonably adequate for this purpose. They do not give satisfactory results with grease because of the cohesiveness of the latter and the fact that the grease does not flow freely. In order to overcome this difficulty, a grease coating device was designed employing the principle of the Doctor Blade in combination with some means to apply light pressure to the grease while the film is being applied to the specimen. In addition, the device was designed to apply films to cylindrical specimens because it is simpler to produce cylindrical rather than flat specimens of the required uniformity. A cylindrical specimen also facilitates measurement of the thickness of grease film and eliminates the edge effect which is encountered in rust tests on flat surfaces.

Grease Coating Apparatus

A schematic drawing of the grease coating mechanism is shown in Fig. 1. The apparatus consists of a conical vessel which contains the grease. Grease films are applied by the mechanism in the following manner: The cleaned steel rod specimen is inserted in the holder, held in place by means of a recessed set screw, and placed in the cylinder so that the opening of the grease cup is blocked by the tip of the rod. The cone is filled with grease nearly to capacity, the cap is put on and the rod is then pushed in all the way in order to prevent the grease from escaping through the hole in the cap (the tip of the rod pushes the excess grease out and the latter is removed). The cap is gradually turned until light finger pressure is applied to the grease. The specimen rod is then withdrawn from the grease with a uniform motion, while simultaneously

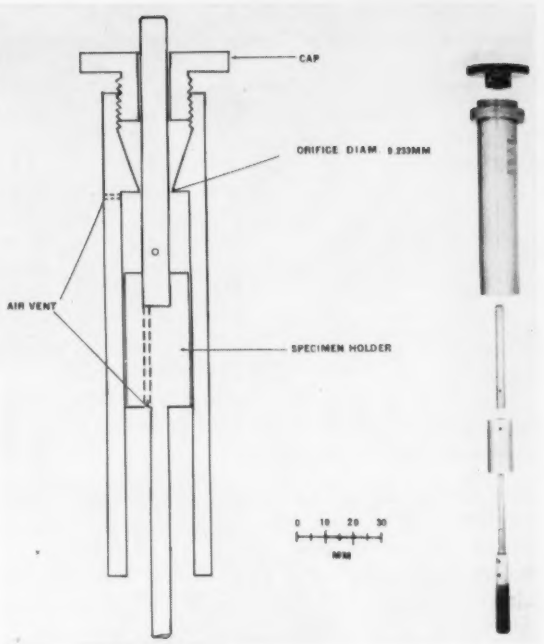

FIGURE 1, grease coating apparatus

uniformly turning the cap of the grease vessel so that a constant pressure is applied to the grease. The specimen is removed from the holder and placed upright in a suitable support. The circumferential uniformity of the grease film on the specimen obviously depends on the degree of concentricity of the specimen rod

TABLE I—GREASE COAT THICKNESS*

Grease No.	180				186				290				399				514				540			
Rod No.	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24
Determination No.																								
1	39	49	61	55	55	64	61	58	42	39	44	41	51	53	46	47	63	59	56	64	61	55	61	62
2	44	47	60	50	70	61	64	68	45	35	48	44	47	57	45	46	64	54	55	62	62	53	54	58
3	49	50	53	55	64	64	70	61	47	39	43	43	49	57	49	50	61	60	61	58	64	59	55	53
4	45	48	55	48	57	70	69	63	37	41	42	38	48	57	48	48	59	60	55	63	60	56	58	58
5	45	53	45	58	63	59	64	55	41	41	37	40	53	51	51	48	62	60	62	57	63	53	60	60
6	41	51	48	55	57	58	61	60	47	43	44	40	50	50	48	50	53	61	60	61	61	51	55	62
7	47	48	52	52	55	63	59	66	51	38	41	43	48	53	48	51	54	55	57	60	55	58	56	51
Mean per col. $\bar{X}$ c:	44	49	53	53	60	64	64	62	44	39	43	41	49	54	48	48	59	58	58	61	61	55	57	58
Mean per grease $\bar{X}$:			50				62				42				50				59				58	
Grand mean $\bar{\bar{X}}$:			54																					
Overall Standard deviation $\bar{\bar{S}}$:			8																					

* All measurements are in microns

TABLE II

Frequency Distribution of Grease Thickness Measurements

Thickness (Microns)	*Grease No.*						*Total*	*Pct.*
	180	186	290	399	514	540		
31-35	—	—	1	—	—	—	1	0.6
36-40	1	—	8	—	—	—	9	5.4
41-45	5	—	15	1	—	—	21	12.5
46-50	10	—	3	17	—	—	30	17.9
51-55	9	3	1	7	6	10	36	21.4
56-60	2	6	—	3	11	10	32	19.0
61-65	1	12	—	—	11	8	32	19.0
66-70	—	7	—	—	—	—	7	4.2
Total	28	28	28	28	28	28	168	100.0
Range (microns)	22	15	16	12	11	13	35	

Total Range 35-70 microns
Average range 15 microns

and the opening of the grease trap. The longitudinal uniformity of the grease film depends on the uniformity of the rod diameter and the fit of the specimen holder in the cylinder in which it lies. The maximum clearance between the specimen holder and the cylinder wall is 6 ± 3 microns.

Film Uniformity

In order to determine whether the films produced were uniform and of approximately equal thicknesses for different greases the following procedure was used. The diameter of the rod was measured to the nearest .0001 inch (2.5 microns). The rod was then coated with the grease and the latter was wiped off a longitudinal segment of the rod. The diameter of the rod plus the remaining grease coat was then measured with a traveling microscope (40X), the film thickness being equal to the difference between this measurement and the rod diameter. This technique is preferable to measuring a completely coated rod since it eliminates errors due to compensating non-uniformities in film thickness of opposite sides of the rod.

Film thickness measurements were made on six greases, using four different rods for each grease. Four initial measurements were made on each rod at approximately ¾ inch intervals. The rod was then cleaned, recoated, and three additional measurements were made at the top, middle, and bottom of each rod. The data are given in Table 1. Numbers one to four represent the first set of measurements and numbers five to seven the second set. The frequency distribution of the data is given in Table 2.

In order to evaluate the effect of variables in method and materials, the data were subjected to an analysis of variance (5,6). The effect of the following variables on film uniformity were studied:

1. Effect of the functioning of the mechanisms, e.g. misalignment, excessive tolerances (referred to as main effect X).
2. Differences among the greases, e.g. texture, consistency (main effect Y).
3. Irregularities in the rod diameters (main effect Z).
4. Interactions between the above (effects XY, etc.)

A significant variation in these effects indicate that the corresponding variable is causing a significant difference in the uniformity of the grease film. Determi-

TABLE III

Analysis of Variance

a. Determinations 1 to 4

Variables	*Sums of Squares of dev.*	*Degrees of Freedom*	*Mean Square Deviations*	*F*	*Significance* (0.05)
X	24.41	3	8.14	0.859	Not significant
Y	5140.45	5	1028.09	108.51	Significant
Z	22.16	3	7.39	0.780	Not significant
XY	161.97	15	10.80	1.140	Not significant
XZ	156.76	9	17.42	1.838	Not significant
YZ	827.22	15	55.15	5.821	Significant
XYZ	426.36	45	9.4747	1.000	———
Total	6759.33	95			

b. Determinations 5 to 7

X	21.00	2	10.50	0.741	Not significant
Y	2949.80	5	589.96	41.64	Significant
Z	18.16	3	6.05	0.427	Not significant
XY	110.83	10	11.08	0.782	Not significant
XZ	41.89	6	6.98	0.493	Not significant
YZ	348.25	15	23.22	1.639	Not significant
XYZ	425.00	30	14.1667	1.000	———
Total	3914.93	71			

TABLE IV
Test Greases

Grease No.	Description	Specification	NLGI Consistency No.
180	Li soap - diester	US Army 2-134	2
186	Li soap - mineral oil	US Army AXS-637	3
290	Bentone - diester	Experimental sample	0
337	Bentone - diester	Experimental sample	2
338	Commercial grease	MIL-G-3278	2
361	Bentone - diester	Experimental sample	1
399	Li soap - mineral oil/diester blend	MIL-G-3278	2
501	Commercial grease	MIL-G-3278	2
514	Silica gel - diester	Experimental sample	2
538	Same as No. 399, plus rust inhibitor	Experimental sample	2
540	Na Soap - mineral oil	MIL-G-2108	2

nations one to four and five to seven were analyzed separately and the results are given in Table 3.

The analysis of the data indicates that variations caused by main effects X and Z and interaction XZ are not significant. This means that the apparatus as constructed is capable of producing uniform grease films. While the data in Table 3 show that different greases (main effect Y) may yield variations in film thickness, this is probably due to differences in texture and consistency. These variations do not, however, affect the uniformity of the film (interaction XY). The effect of differences among greases is limited by the physical characteristics of the apparatus, for there exists an upper limit for the film thickness and this depends on the total clearance available, i.e. the difference between the cup opening and rod diameter. It is possible to produce a grease film the thickness of which slightly exceeds this total clearance. This can be done by exerting extra pressure on the grease during the coating procedure. The data show, however, (Table 2) that this occurred in less than five per cent of the cases. In the model used, the orifice diameter was 9.233 ± .002 mm, and the measured rod diameter range was 9.100 to 9.134 mm (the median being at 9.125), thus allowing a maximum of 67 ± 2.5 microns for the grease film. It shsould be pointed out (Table 4) that the test greases were selected to cover the consistency range normally associated with instrument greases, from grease No. 186 with a penetration value of less than 250 to Grease No. 290 with a consistency bordering on a semifluid system, and which may almost be applied by dipping.

Rust Tests

In order to test the reproducibility of the method, rust tests were conducted on a number of greases. Their compositions (Table 4) included mineral oil and synthetic base fluids, soaps and nonsoap gelling agents, and the presence or absence of rust inhibitors.

The specimens were prepared in the following manner: The specimen, a 4 inch X-1020 steel rod, is wiped dry with a lint-free cloth and polished with 3/0 and 5/0 sandpaper. It is then submersed in boiling benzene (thiophene free) for approximately 3 minutes, removed, and placed in boiling petroleum ether (BP 30-65° C.) for another three-minutes. The application

TABLE V
Rust Tests

Grease No.	Mean Film Thickness, Microns	Rust Formation, Hours A	B	C	D	Mean Hours	Stand. Dev. Hours	Stand. Dev. Per Cent
180	50	900	1000	940	940	945	41.23	4.36
186	62	1400	1280	1324	1400	1351	59.37	4.39
337	46	144	196	240	204	205	40.94	20.0
338	53	1400+	1400+	1400+	1400+	1400+	....	....
361	64	120	120	144	144	132	4.38	3.32
399	50	306	384	336	306	333	36.83	11.06
501	56	1400	1244	1244	1400	1322	45.03	3.41
514	59	126	154	154	147	147	4.24	2.88
538	58	1160	880	1024	1100	1041	120.9	11.61
						AVE.		7.63

of the grease film is then carried out as previously described. The specimens were then placed in a humidity cabinet and exposed to the following cyclic conditions: Four hours at 80 per cent RH and 110° F., followed by four hours at 95 per cent RH and 130° F. The transition from the low to the high conditions produces a controlled condensation period. Four specimens were used per grease and the tests started at the low conditions. The appearance of one rust spot, one mm or larger, in diameter, which grows perceptibly over a period of 24 hours, or two or more rust spots of any size were taken as the time of failure for the specimen. (The relative severity of the conditions in this cabinet is considerably greater than that of the conventional 100 per cent humidity cabinets. Limited comparisons were made with the JAN-H-792 humidity cabinet, using oils. These tests indicated an approximate severity factor of 2.5). The results of the rust tests are in Table 5. The data show that the level of reproducibility is essentially the same regardless of the duration of the test. The overall reproducibility of the test expressed by the standard deviation as a fraction of the mean is 7.6 per cent. The data further show that this test is capable of distinguishing not only between good and bad greases but also between good and better greases, which is most desirable in an evaluation of a rust inhibitor or the effect of inhibitor concentration.

Acknowledgement

The authors wish to express their appreciation to the U. S. Army Ordnance Corps for permission to publish this paper.

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About the Authors

K. R. Fisch *graduated in* 1949 *from Temple university with an AB degree. Since* 1948 *Mr. Fisch has worked in the chemistry research laboratory of the U. S. Army Ordnance Corps, Frankford Arsenal, in research and development of instrument lubricants, additives and development of related testing equipment. He served as a consultant to the U. S. Army Signal Corps on lubrication problems. He is a member of ACS.*

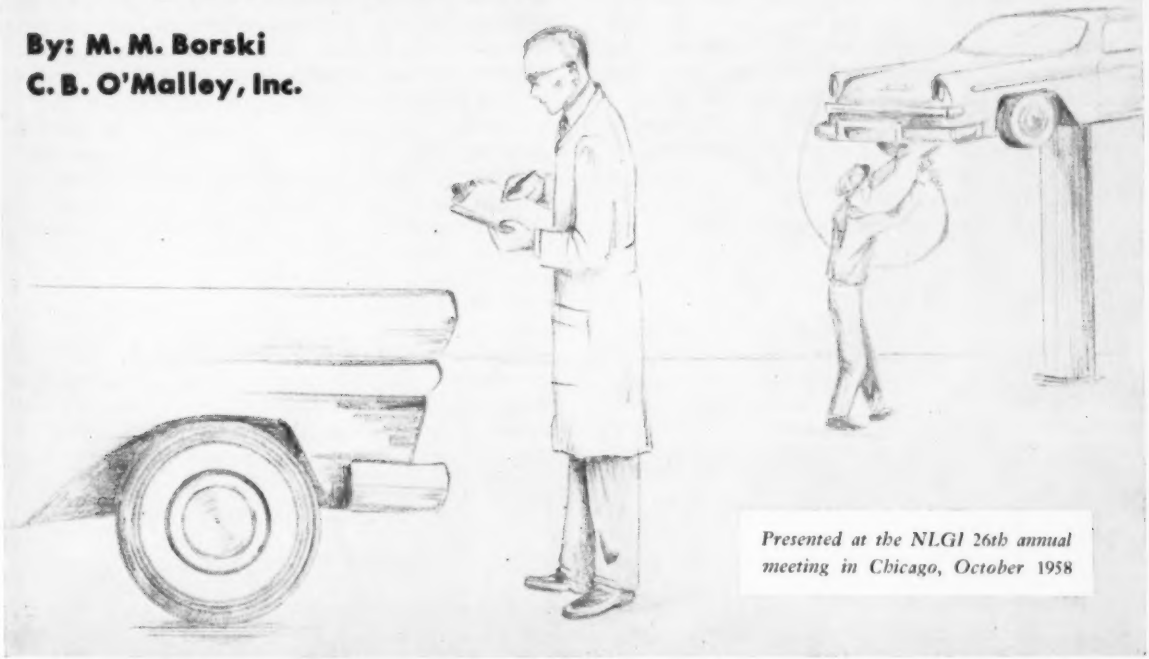
H. Gisser *received a BA degree from the City college of New York in* 1936, *an MA degree from Columbia university in* 1937 *and a PhD from Temple university in* 1952. *He has been associated with Pitman-Dunn laboratory, Frankford Arsenal since* 1939. *At the present time he is director of the chemistry research laboratory, Pitman-Dunn laboratories. Mr. Gisser's publications include various technical articles on lubricants, additives, oxidation, and other related subjects.*

J. Messina *graduated from St. Joseph's college, Overbrook, Pa. in* 1934 *with a BS degree. After graduation he spent five years as a materials analyst at the Pennsylvania department of highways testing laboratory. Harrisburg, Pa. He joined the U. S. Army Ordnance Corps, Frankford Arsenal, Philadelphia, Pa. in* 1941 *and became associated with research and development on lubricants for Army Ordnance instruments. His current position is chief of the lubricants unit, chemistry research laboratory. His publications include technical articles on additives and non-spreading oils.*

From the Car Dealer's Viewpoint—

How to Get the Motorist to Lubricate Every 1,000 Miles

By: M. M. Borski
C. B. O'Malley, Inc.



Presented at the NLGI 26th annual meeting in Chicago, October 1958

AN OLD tried and true way to get customers to lubricate at 1,000 mile intervals is to never let a customer get out without a lubrication once he has set foot in for another service, and the records or sticker say he needs one. This is still good salesmanship, although it smacks of "barber-shopping" a customer. This is a type of "selling" which is opposed to educating a customer, which is the basis of our problem. Customer education cannot be accomplished by salesmanship alone, but can only become a fact when all areas of retail merchandising come to play on the objective.

In our buyers market, a high degree of sales resistance has been developed in the thirteen years since the end of the war. Customer's dollars are sought after in so many different ways that a subconscious wall goes up as soon as the first sign of a pitch is made. However, if he is conditioned properly and comes to the decision to make a purchase by himself, everyone's job is a lot easier. This conditioning process is the backbone of successful retail merchandising in this merchandising-minded decade that we live in. It is considerably more difficult to achieve than merely asking for the order through the "hard sell" formula. This conditioning process is the key to merchandising—a key that hundreds of millions of dollars are spent on each year, and I don't believe that a universal solution will ever be found—as none exists. A key is a very individual thing, designed to fit one lock and only one lock. Therefore, the things that will condition one customer will not condition another. The high-priced hucksters that infest the advertising agencies of Michigan Avenue here in Chicago will tell you, in all sincerity, that advertising is the universal answer. The high-pressure drummers that peddle gimmicks to unsuspecting retail merchants will all but "guarantee you" success. Oil salesmen, God love them, will tell you that their brand of oil has a magnetic attraction that will make you independently wealthy. All of the foregoing are factors

that, each in their place, have a role in merchandising that conditions the public, but as soon as you concede that one is your trump card, you admit defeat.

The service department of an automobile agency is interested in lubrication business because it is profitable, but this isn't a very good sales story to the customer. We have 33 people on our payroll and a good lubrication business is one way of reducing the overhead. We want to lubricate every car that comes in. But a car dealer does not get the exposure to the customer that the men in the service station do. When the writer worked at a service station, a good customer came in about twice a week for gas, and we got to know them and their requirements very well. They come to an automobile agency only three or four times a year. We, therefore, have to depend on"mechanized" selling a great deal to take the place of personalized selling as best it can. Since we do not have the personal contacts, we have to send out costly mailers, use specials and "loss-leader" promotions, et cetera. We have to try and wed the customers to our place of business. To accomplish this, we feature parts warranty, which is a warranty extended by an oil company which agrees to replace lubricated parts for the length of the warranty period—usually 25 to 35 thousand miles. For this "parts warranty" to stay in effect, the owner must lubricate and change oil at least every 1,250 miles—and in *your* place of business. This is one of the many devices designed to instill in the customer's mind the desirability and value of bringing his car "*home*" for service. And when he does, we have a tailor made opportunity to sell him a lubrication job.

We have yet another selfish reason for bringing a customer's car to us for service of any kind. We want to *make sure* that the car is *serviced properly* so that they will be so satisfied with its performance, that they will buy another car from us in one, two, or three years. And talking about keeping the customer satisfied, a point that shows how important a part lubrication plays in that satisfaction, we want to mention the front end ball joints on some new models,. These ball joints begin to "grunt" pretty bad when they are not properly lubricated. The customer hears that grunt—he brings his car in—we lubricate it and the grunt goes away. It's an understandable example to the customer of the importance of regular lubrication. The ball joint "grunting" that results from improper lubrication can reflect on the car and hurt the chances of our selling this customer another car in the future.

Regarding oil changes, many customers have the strong belief that they can drive thousands and thousands of miles without changing the oil and to them it still looks good. It is difficult for them to understand that even though it might look the same, it doesn't have all of its original properties, hence it is worn out. To change this thinking required education, not hard sell, obnoxious salesmanship. Many auto manufacturer's manuals recommend oil change periods of from 500 to 3,000 miles—depending on the type of driving the individual motorist does. Many motorists state, "Oh, I never use my car under extreme conditions; never abuse it, so why should I change oil every 1,000 miles? Why, we only use it for short trips to work, to the store and etc." Actually it is usually not too difficult to point out to a motorist that short trip driving is the hardest type of service his unit can be subjected to, explain to him that practically all wear in a modern engine occurs during the start and warm-up period, that sludge and carbon deposits build up much more rapidly this way than from high speed highway driving. These tiny bits of carbon are actually abrasives, the only way he can rid his engine of these harmful abrasives is by changing his oil. After motorists have been educated to these facts and ideas, selling these services to them is almost automatic.

The cars that are sold in our agency cost as much as I used to make in a year, and it wasn't too many years ago either. A $4,000.00 automobile next to the price of a lubrication is a pretty big thing and a good sales tool —a tool that utilizes the safety approach. Our agency uses this comparison very effectively, as we service some 45 cars for something daily. Of this number, we lubricate an average of 30 which is considered pretty good.

Customers, when they are confronted with a technical problem, like to have the confidence of the men making the recommendation. If this confidence and trust isn't there, you are just another garage. We work hard on getting this confidence—not in words, but in actions.

How do you develop this trust? That's a good question. How can a guy like me achieve the same type of trust as a professional man, such as a doctor or dentist has? When a doctor tells you your kid has to have his tonsils out, you take his word as you trust him and you believe that he isn't merely trying to beat you out of a fee. As a "doctor of motors", our "patients" have to have confidence in us so that when we tell them they should have their cars lubricated every 1,000 *miles for safety*, they'll believe it and get it done.

The manufacturer's recommendations can be a good sales tool, or it can be a liability. For instance, the owners manual of the type of car that we service most in our shop, recommends that the car be lubricated every 2,000 miles. Other makes are now using a more realistic figure of 1,000 miles. When we say that "our experience has shown us that 1,000 miles is better," or if we say "we have found that 1,000 miles is better," the customer has to respect our judgment; he has to have confidence in us.

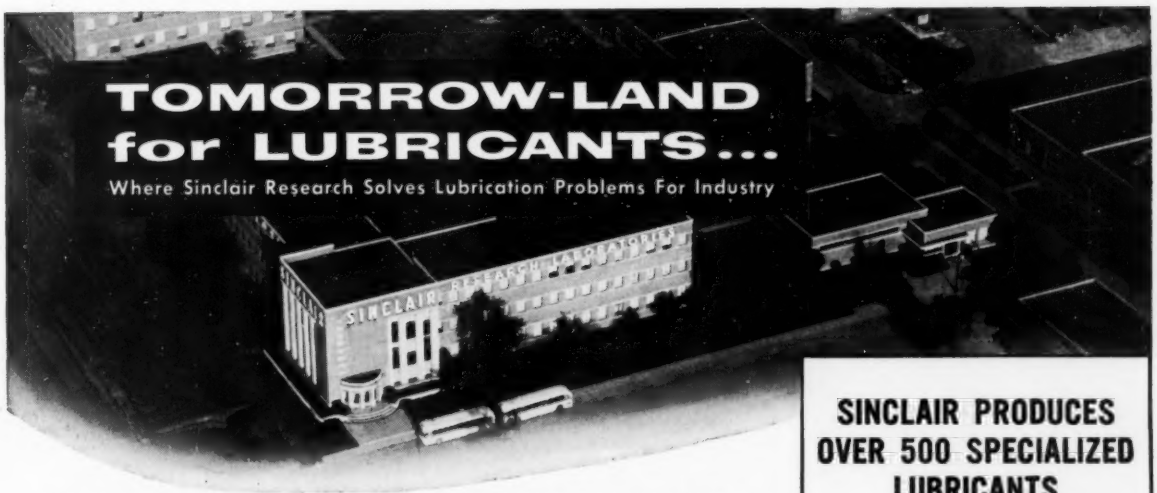
Lubrication, when properly done, is more than merely hitting a few fittings. It provides an opportunity for the service man to check many important areas that he wouldn't normally have the opportunity to. Encourage the customers to watch the work being performed. About *one half our customers* do and they're impressed by the completeness of the work. This sells lubrication. A clean, well kept shop is important if you

hope to develop the respect of your customers. This sells lubrications.

Admittedly, this has been a group of personal experiences rather than a bag full of tricks. There is no bag full of tricks, or pat formula on how to get your customers to lubricate every 1,000 miles for safety. There are a number of sound merchandising principles that have worked for us; perhaps they could work for others. And finally, I am sure of one thing that would help more than anything else, and I've saved it till the very last. The "lubricate every 1,000 mile" idea is going to take root only in isolated cases here and there, and only while the individual management keeps pressing. The idea will never take hold all over, as it must to produce the values that I think it has, until the car manufacturers stop recommending long interval lubrication periods and oil company engineers stop pussy-footing and saying that their oil lasts forever. I know that just recently a paper was read stating that *oil does wear out*. That's only one man, one paper, and it just happened yesterday. So it doesn't mean too much yet. The majority of oil company technical people are still not ready to accept the value of 1,000 mile lubrication because if they were, they would do a better job of convincing the car manufacturers. So I could wind up this talk by putting the title of my talk in the form of a question: "How Can We Convince The Motorist To Lubricate For Safety Every 1,000 Miles?" The answer in the main, in my opinion, would be: "Let's start with you! And I hope you start."

About the Author

M. M. Borski *started out in the automobile business as a service station attendant. Later he became a lubrication man for a new car dealer and worked his way up to mechanic, shop foreman, assistant service manager and finally service manager. During the period 1956 to 1958 he was employed by the Oldsmobile division of General Motors as a Service representative in the Chicago metropolitan area. Mr. Borski is married and has one child and has served three years in the U. S. Army. He currently holds a position as service manager for C. B. O'Malley, Inc., one of Chicago's leading Oldsmobile dealers.*



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New Quarters for ILC

International Lubricant corporation, manufacturers of lubricating greases and motor oils since 1929, has moved its executive offices and laboratory facilities into a handsome, new $850,000 two-story building of contemporary design on its 23-acre tract just northwest of the city limits of New Orleans on the Airline highway.

The move into the 21,000 sq. ft. structure of reinforced concrete was made primarily to bring together all of ILC's extensive laboratory and testing equipment, much of which had been scattered throughout the plant on the same property since the destruction of the former lab by fire on December 13, 1955.

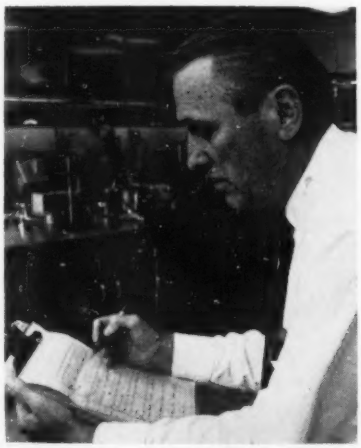
In addition to the unified laboratory facilities, which occupy triple the space of the old lab—about 6,400 sq. ft. in all on one floor, or the entire south wing—the new building contains executive and sales offices for ILC, which makes lubricating greases and oils for automotive, marine, aviation and industrial uses.

Research Continuing Program

"We have always done our best to develop and manufacture the finest greases we could and to carry on maximum research in this field," William H. Saunders, Jr., founder and president of ILC, said on the occasion of the move. "We regard the new laboratory here as the most important part of that continuing program."

"This program is under the direction of Harold M. Fraser, our vice president in charge of manufacturing, research and development, and it has enabled International Lubricant to pioneer in the

FOUNDER and president, W. H. Saunders, Jr., with company seal in back.



VICE president of manufacturing and research for ILC, Harold M. Fraser.

TWILIGHT view of ILC lab, which houses more than 20 chemists in modern testing and research facilities, just out of New Orleans.

production of a lithium 12 hydroxy stearate as a carrier, a kind of 'global grease,' and to develop a calcium hydroxy stearate grease with an unusually high degree of corrosive resistance and mechanical stability."

The 20 chemists who make up Fraser's staff use laboratory facilities that are divided into four sections—one for oil testing, one for grease testing, one for research, and the pilot plant. Recognized oil and grease testing facilities are supplemented by ILC's wet- and dry-oscillating friction machines and the heated roll test, as well as other laboratory equipment.

Other features of the lab, which Saunders regards as one of the most extensive in the lubricant processing field, are a walk-in cold room where greases are tested at 40° below zero Fahrenheit; a uniform temperature room (77°F); a flash and fire room; a dark room with a microscope, a spectroscope and an electronic colorimeter, and a comprehensive reference library.

Sharp Contrast with Old

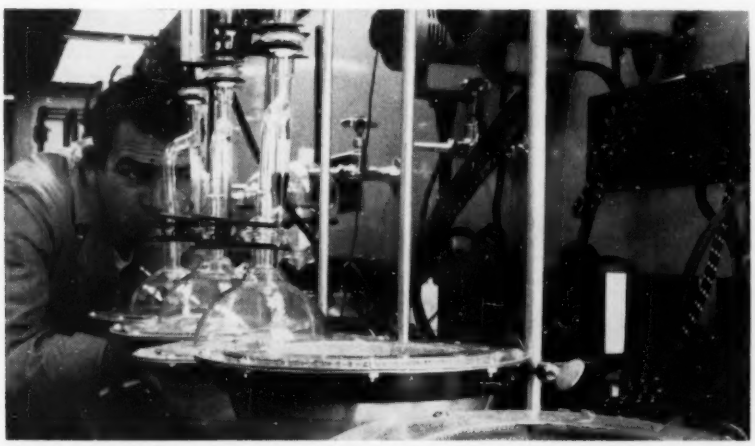
The new lab is in sharp contrast to the 300 sq. ft. assigned to Fraser when ILC began its operations 30 years ago in a two-story steel building with a total floor area of just 12,000 sq. ft. on a ⅝-acre plot that is the heart of the present property. Fraser had to do most of the testing himself, and to maintain the production line.

The rather primitive, if effective, production facilities have been radically changed, too. Today ILC has a grease plant with an approximate capacity of 90,000,000 lbs. a year, a modern oil compounding plant for handling, storing, blending, filling and canning as much as 350,000 to 400,000 gallons a day; barge unloading facilities on the Mississippi River with two 8-inch pipe lines to its tank farm where 7,000,000 gallons of oil can be stored.

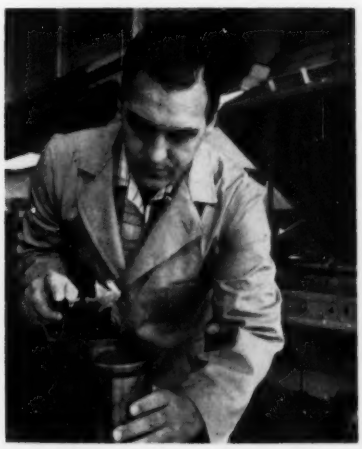
In 1946 ILC was purchased by the Shell Oil company, one of its best customers for the ten years preceding the change in ownership. Saunders and Fraser continued as the operating officers, and last year Benjamin G. Symon, who had been manager of the industrial products department for Shell in New York, was added to the management team as vice president of sales.

SALESMAN on the job . . . B. G. Symon, just made vice president, sales.

(Saunders is a former president of the Institute, a director since 1933, and the one founder of NLGI still active in the Institute. Symon is a former president and director, having served since 1944 until his retirement from the Board last year. Fraser has long been active in Technical Committee affairs, and is one of the first recipients of NLGI's "Award of Achievement.")



QUALITY control in the new laboratory with chemist R. W. Beron checking water determination in each grease batch.



PACKING cylinder, technician Joseph Steinkamp readies heated roll test.

Literature and Patent Abstracts

Processing

Fused Metallic Soaps in Lubricating Greases

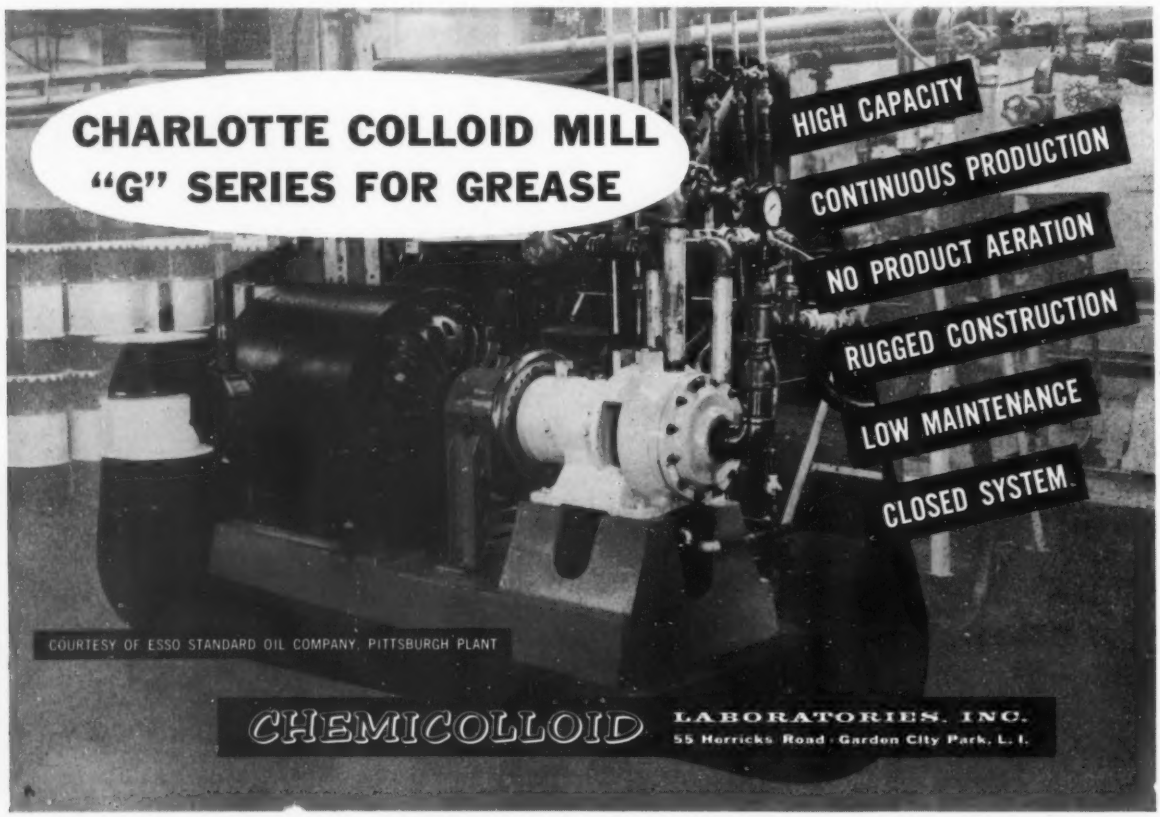
Renshaw and Lamson (U.S. Patent 2,861,950) describe the production of lubricating greases in which metallic soaps are fused, cooled, mixed with lubricating oil and milled at room temperature.

Thus, lithium stearate was fused and four grams of this soap was ground to pass a 28 mesh screen and be held on a 42 mesh screen. After mixing this soap with 21 grams of a lubricating oil having a viscosity of 65 SUS at 210°F., and passing through a three roll paint mill three times a lubricating grease resulted having 16 per cent soap and a micro penetration of 88.

Method of Grease Manufacture Comprising Shearing

A limited amount of shearing is recommended in the case of the manufacture of lubricating greases thickened with lithium hydroxy stearate. For this purpose Pitman, Hordan and Bright (U.S. Patent 2,870,090, assigned to the Texas company) pass a cooled mixture of the soap and oil through a shear valve with a pressure drop across the valve of 15 to 200 psi. The shearing employed is represented by an amount of work expended upon the grease of 50 to 500 pounds per pound of product.

By the use of such a method of shearing the soap content for a given grade of lubricating grease is greater than when milling is used. For example a mixture was made containing six per cent of lithium soap of 12-hydroxy stearic acid which, after melting at 395 to 405°F., was cooled by recycling through an external cooler. Portions of the cooled mass were withdrawn from the kettle through a shear valve adjusted to a 65 or a 90 pound pressure drop. Another por-



tion was passed through a Premier colloid mill adjusted to 0.003 inch clearance. The following table indicates the effect of various methods of treatment on penetrations.

TABLE I

Finishing Method	*ASTM Penetrations 77°F.*	
	Unworked	*Worked*
No Shearing	400+	379
Colloid Mill Treatment	288	315
Shear Valve, pressure drop		
65 psi	370	366
90 psi	356	357

Application

Life-Lubricated vs Conventional Bearings

W. A. Williams, *Power Transmission Design*, February 1959, p. 45-46.

Consideration is given to the reasons why one or the other of the above types of bearings should be selected for use or installation. A larger part of such reasons for selection are concerned with lubrication and therefore are of interest to our group.

It is stated that the bearing manufacturer will not take a chance on using a cheap lubricating grease for a life-lubricated bearing. Further, the right amount of such lubricant will be used and this will be applied under ideal conditions so that contamination and dirt will not be introduced. Since the sealed-for-life bearing is built to be free of most of the human elements of maintenance, it is desirable in appliances and other products where maintenance is impossible. This type of bearing uses grease only.

The conventional bearing, which can be relubricated at will, has a number of advantages. For lightly loaded bearings, at normal speeds the life of the lubricating grease is normally shorter than the bearing life and thus there is an advantage in being able to replace the lubricant at regular intervals. Under conditions of high speed the life of the lubricating grease is still shorter and therefore permanent lubrication is not practical. The same is true of bearings operating at temperatures of 250°F. or above since few lubricating greases will stand up under such conditions and hence must be replaced frequently.

Testing

Grease Testing

J. K. Vose, *Scientific Lubrication* 11, No. 2, p. 14-17, 1959.

The author, who is connected with the Motor engineering dept., Metropolitan - Vickers Electrical Co., Ltd., states that much of the credit for the increase in both numbers and applications of electric motors over the past fifty years is due to the successful adoption of the grease-lubricated rolling bearing. The availability of suitable lubricating greases is of such importance to manufacturers of motors that investigations have been made into the performance of various types of such products.

By the early 1930's a high-quality soda base grease was accepted as the most suitable for this service. It was considered that such a lubricant functioned by "weeping" of oil from the grease close to the bearing. To determine the rate of oil-release from the lubricant an oil-release test was devised (see *Scientific Lubrication*, December, 1958). Such a test was therefore made a part of the purchasing specifications. However, from 1939 to 1945 and even thereafter there was a deterioration in the performance of available lubricating greases.

This led to the development of the M-V grease-testing rig (see *Metropolitan-Vickers Gazette*, December 1956). This makes use of bearings of three inch bore diameter operating at 3,000 rpm.

Tests to date indicate that lithium base lubricating greases are superior to soda and soda-lime greases previously used. In fact the excellent performance of such lubricants poses a difficulty in the length of time required for a test, in fact the author states: "With the best lithium greases tested so far, only one test per annum can be completed on each rig."

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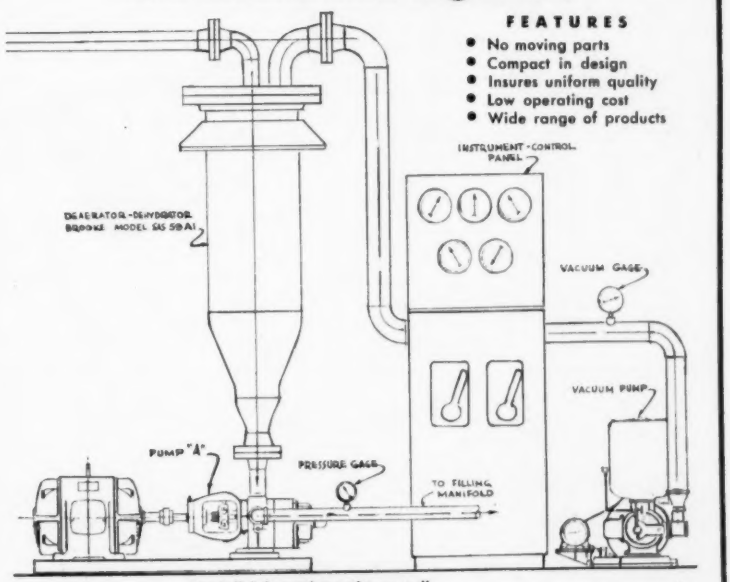

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SERVICE

Open Gear Lubricants

According to McGrogan (U.S. Patent 2,870,089, assigned to The Atlantic Refining Co.) open gear lubricants which have high dropping points and still remain plastic and tacky at low temperatures consist of a small amount of sodium soap formed in situ in a residual steam refined asphalt.

The recommended asphalt is a No. 2 saturant grade which is partially oxidized. Such a typical product had a penetration at 77°F. of 293, a viscosity Saybolt Furol at 300°F. of 42, and a flash point of 475.

The preferred fatty acids used are those of hydrogenated fish oil. A typical lot of the gear lubricant started with the addition of 5718 pounds of No. 2 saturant grade asphalt in a kettle equipped with a "lightning" mixer, a recycle gear pump and an air blower. All three of these mixing devices were used and the charge heated to 150 to 190°F. before 150 pounds of hydrogenated fish oil fatty acids were added. Next the temperature of the mass was increased to 190 to 195°F. and a 25 per cent solution of sodium hydroxide, containing 21 pounds of the solid alkali, was added. After two hours the temperature was increased to 225 to 230°F. while the mixing was continued. Air blowing was intermittently stopped for the incremental addition of 152 pounds of graphite. Finally 61 pounds of methyl lardate was added and mixing, recycling and air blowing continued at a temperature of 230°F. until the lubricant had an ASTM dropping point of 360°F. Heating and air blowing were discontinued and 10.9 weight per cent of Stoddard Solvent was added at the suction side of the recycle pump.

The final product had an ASTM dropping point of 378°F. and an unworked penetration at 77°F. of 343. The product was stated to have extreme pressure qualities.

In general 0.88 to 4.39 per cent of the fatty acids are used in forming the lubricant. The Stoddard Solvent used can be increased or omitted. In the latter case up to fifteen per cent of a 100 viscosity SUS at 100°F. can be included in the final gear lubricant. Likewise either the graphite or the methyl lardate or both can be omitted from the composition.

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Gear Lubrication

Lubrication Problems in High-Speed Gears

Jack W. Dern, *Lubrication Engineering*, 15, p. 23-27, 1959.

Problems connected with lubrication of spur gears operating at pitch line velocities of 20,000 to 25,000 feet per minute are discussed. It was found that gas turbine oil, specification MIL-L-7808 proved to be a satisfactory lubricant for the gears in question.

This oil was sprayed by jets on the leaving side of the mesh. Scav-

enged oil flow from both gears and bearings required design improvement so that removal would use up some of the kinetic energy of the oil.

Compositions

Lubricating Greases Thickened With AZO Compounds

Lyons and Odell (U.S. Patent 2,851,418, assigned to The Texas company) use fifteen to 35 per cent of high melting azo pigments to thicken lubricating fluids, such as mineral oils, esters and silicone polymers, and form lubricating greases which are useful for high temperature bearing applications.

Milling of the pigment with the fluid forms the lubricant. For example, 25 per cent of azo pigment was mixed with 71 per cent of a fluid consisting of 7.1 per cent mineral oil and 92.9 per cent of a synthetic ester. One per cent of diphenyl-p-phenylene diamine was dissolved in 3 per cent of tricresyl phosphate and added to the above mixture after which the total mass was passed through a Premier colloid mill. This gave a lubricating grease with a worked penetration of 283 which ran 210 hours in a high temperature bearing test at 400°F.

Lubricating Grease Containing A Boron Ester

Dykstra (U.S. Patent 2,862,879, assigned to Ethyl corporation) makes use of esters of metaboric acid to remove nitrogen from petroleum hydrocarbons. In one example, 318 parts of a toluene solution of isopropyl metaborate containing 13.2 parts of boron were reacted with 32,900 parts of a neutral lubricating oil of 31.1° API gravity and a viscosity of 125.4 SUS at 100°F. The oil contained 0.0029 per cent basic nitrogen before treatment. After the above mixture was stirred for 11 hours at 50°C. and allowed to settle at room temperature for 31 hours a gelatinous precipitate was removed by filtertion.

Analysis of the oil after filtration showed a basic nitrogen content of 0.0011 per cent and also that substantially all of the boron remained in the oil. Eighty-eight parts of this oil were blended with one part of lithium stearate and eleven parts of aluminum stearate to make a lubricating grease which was stated to be of superior quality. No other information about the lubricant is given.

Hydrous Metal Salts as Thickeners For Lubricating Greases

According to Morway (U.S. Patent 2,863,847 assigned to Esso Research and Engineering Co.) lubricating fluids can be thickened to grease consistency with five to 30 weight per cent of a hydrous mixture of a metal salt of a low molecular weight acid and a metal salt of an intermediate molecular weight acid. The low molecular weight acids include formic, acetic, propionic, acrylic or lactic. The intermediate molecular weight acids include those having seven to ten carbon atoms per molecule with caprylic being preferred. The ratios of low to intermediate molecular weight acids should be between two to one and 40 to one.

For example, 66.7 parts of a mineral lubricating oil having a viscosity of 80 SUS at 210°F. and 12.8 parts of hydrated lime were charged to a kettle and mixed to a smooth slurry at about 70°F. A mixture of eighteen parts of glacial acetic acid and 2 parts of commercial caprylic acid was then added to the kettle resulting in an immediate reaction which raised the temperature of the mass to about 190°F. where a solid product

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formed. Stirring was continued until the lubricating grease cooled to about 100°F.

The smooth lubricating grease formed above had the following penetrations: unworked 220, 60 strokes 252, 75,000 strokes 319. The product carried a 50 pound load on a Timken test. While other metals can be employed to form the thickener, the alkaline earth metal compounds exhibit outstanding load carrying characteristics.

Herbicidal Grease Composition

A grease is described by Bartlett, Brugmann and Morway (U.S. Patent 2,867,519 assigned to Esso Research and Engineering Co.) which is effective for killing poison ivy, poison oak, weeds, brush and trees. This consists essentially of what would normally be a lubricating grease containing 0.5 to ten weight per cent of a metal salt of 2,4-dichlorophenoxy-acetate.

Thus, five parts of 2,4-dichlorophenoxyacetic acid and fifteen parts of hydrogenated fish oil fatty acids were mixed with 75.7 parts of a hydrofined coastal type petroleum distillate having a viscosity of 55 SUS at 210°F. After the mixture was heated to 150°F., 38 parts of a ten per cent aqueous solution of lithium hydroxide was added. The mass was heated to 400°F. with stirring and 0.5 part of phenyl alpha naphthylamine added before pouring into pans to cool. The cold grease cake was homogenized to yield a smooth, adhesive product having a worked penetration of 275 and a dropping point of 360°F. The product was not soluble in boiling water and hence would not be displaced by rain after application.

The grease described can be diluted with regular crank case oil or even with drainings to the point where it can be sprayed. While 0.5 to two per cent of the floricidal chloroacid compound are frequently sufficient, the preferred amount is two to eight per cent.

If the herbicide is to be dispensed from metal applicators, the addition of corrosion inhibitors, such as

sorbitan monooleate, to the mixture is desirable.

Composition and Process

Anhydrous Calcium 12-Hydroxy Stearate Lubricating Greases

Production of grain-free, substantially anhydrous lubricating greases, in which the thickener is obtained by reacting lime with commercial 12-hydroxy stearic acid, is described by Dilworth Culnane and Nelson (U.S. Patent 2,862,884 assigned to The Texas Co.),

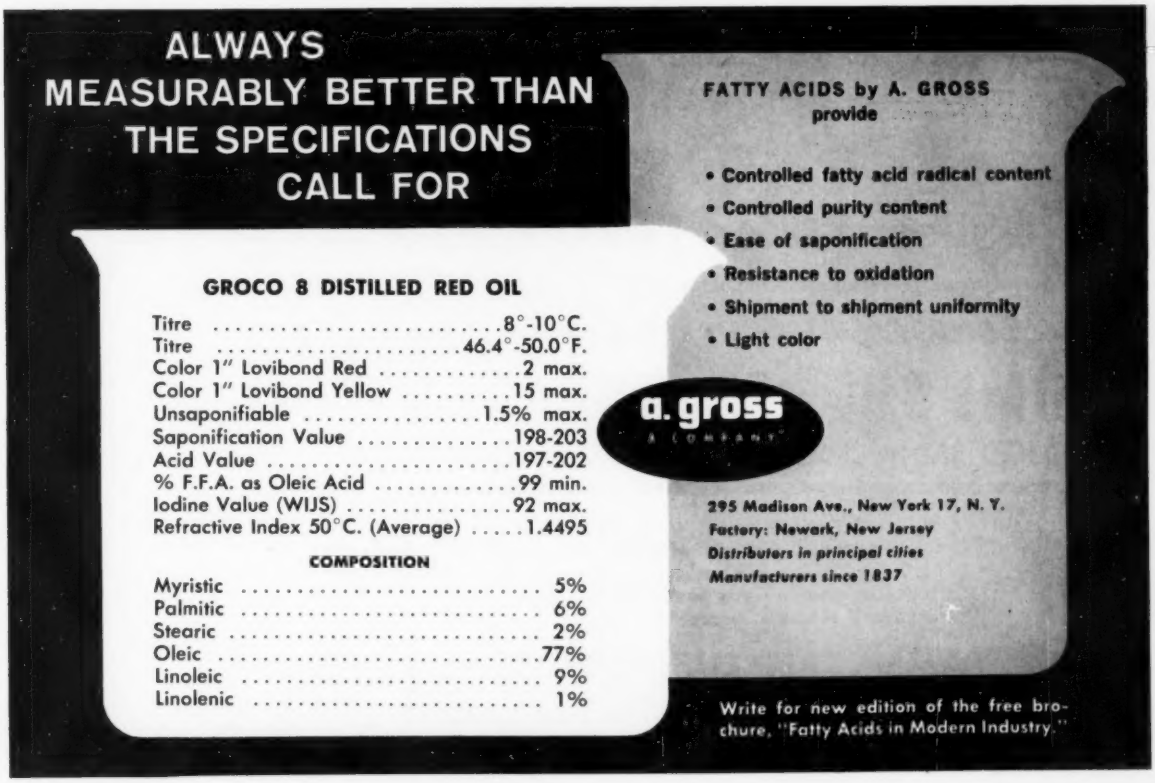
To obtain lubricating grease of the character described, it is stated to be necessary that the hydroxy fatty material contain at least three per cent by weight, and preferably four to ten per cent, of estolides. These estolides are intermolecular esters and polyesters formed by the reaction of the hydroxy group of one molecule of the hydroxy fatty acid with the carboxy group of another.

Further, the presence of a small proportion of naphthenic distillate lubricating oil having a viscosity of 40 to 600 SUS at 100°F. is necessary during the saponification step. The proportion of this oil to the hydroxy fatty acids used should be 0.5 to one to about five to one.

The proposed method of manufacture of the subject lubricating grease consists of reacting the hydroxy fatty acids in the presence of the naphthenic oil at 280 to 330°F. for a time sufficient to dehydrate the mass after which additional oil is added as the mixture is stirred and cooled to about 200°F. A variation of the processing consists of reacting the major portion of the lime at 150-210°F and adding the remainder of the lime at about 270°F.

Thus, a kettle was charged with 9.43 pounds of a naphthenic oil having a viscosity of 59.4 SUS at 100°F., 9.43 pounds of Hydrofol acids 200, 6.5 pounds of water and 629 grams of dry powdered lime. The Hydrofol acids 200 contain about five per cent of estolides. The charge was heated with stirring to 182 to 197°F and held at this temperature for four hours after which it was brought up to 300°F. in about 1.3 hours and finally held at 303 to 309°F. for an additional half hour. With the heat cut off, additional oil was fed in while the mass cooled to 176°F. during about twelve hours. At a temperature of 190 to 200°F. 1352.5 grams of sorbitan monoleate (Span 80) and 227.3 grams of Ortholeum 300, dissolved in a portion of the oil were added together with 450.8 grams of Manvel residuum having a viscosity SUS of 731 at 210°F. The finished lubricant, at 170°F., was pumped through three 60-mesh screens to give a smooth, buttery product.

The resulting lubricating grease contained 10.1 per cent soap and 0.1 per cent of water. The dropping point of the product was 279°F. and the worked penetration 290. Such a product is said to meet the specifications of MIL-G-10924.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People in the Industry

Dr. E. H. Bluman Retires as Head of Harchem Division

Wallace & Tiernan Inc. of Belleville, N. J. announced the retirement of Dr. Ernest H. Bluman as president of its Harchem division effective April 30, 1959. The Harchem division is a prominent manufacturer of industrial chemicals including plasticizers, sebacic acid and fatty acids. Dr. Bluman has guided Harchem's operations in these fields since Wallace & Tiernan's acquisition of W. C. Hardesty company and Hardesty Chemical company early in 1955. Dr. Bluman will continue his association with Harchem in a consulting capacity.

Dr. Bluman was formerly executive vice-president of W. C. Hardesty company, Inc. of New York. He was associated with this organization since 1940.

Educated in Germany, Dr. Bluman received the degree of Doctor of Engineering from the Institute of Technology of Berlin-Charlottenburg in 1920. For a number of years he was active in the fatty acid and related chemical industries in Europe. He served the well-known German chemical firm, Vereingte Stearinwerke of Hamburg, as managing director. He is Harchem's NLGI Technical representative.

Emery Promotes Dr. Charles G. Goebel

The promotion of Dr. Charles G. Goebel to the position of research director of Emery Industries, Inc., has been announced by R. W. Van Tuyle, vice president of manufacturing, research and development.

Other promotions of research personnel are:

Victor J. Muckerheide to technical coordinator: Dr. Karl T. Zilch to succeed Mr. Mucherheide as manager of the process research section: Dr. Louis M. Wise to manager of the Sanitone Research section: and Dr. H. Fred Oehlschlaeger and Dr. Fred O. Barrett as managers, respectively, of the newly created Derivatives Research section and Polymerization Research section, dividing the activities of the former Organic Chemicals Research section.

Dr. Goebel is well known as the discoverer of Emery's process for producing dimer acid by the thermal polymerization of unsaturated fatty acids. As research director, he will manage Emery's expanding research activities on fatty acids and their derivatives.

Staff Changes at Joseph Dixon Crucible Co.

New staff changes at the Joseph Dixon Crucible company, Jersey City, N. J., have been announced by Frank G. Atkinson, president.

H. Edward Ehlers and Warren A. Zimmer, already vice presidents and veteran executives of Dixon, have been elected senior vice presidents, while Sherwood B. Seeley, technical director, was elevated to vice president in charge of research.

Mr. Ehlers, a graduate of Lehigh university, joined the Dixon company in 1935 and became manager of the crucible and refractories division in 1948. Six years ago he was named industrial sales manager and

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a year later he became general sales manager. In 1956 he became vice president in charge of sales.

Trained as a ceramic engineer, Mr. Zimmer was graduated from Ohio State university in 1935. After acquiring his master's degree, he joined Dixon's research department. His career was interrupted by four years of service in the Army field artillery, from which he emerged as a lieutenant colonel. Mr. Zimmer became general superintendent of the crucible division in 1950 and works manager a year later.

Mr. Seeley was a teen-aged scientist. At the age of fourteen he discovered, through an experiment in chemistry, a new way to ignite thermite and won acclaim in scientific journals. He was graduated from New York university with a degree in chemical engineering and went directly from school to the Dixon company. Mr. Seeley is a frequent lecturer at university and professional seminars.

ASLE Officers Named

The 1959-60 national officers and directors of the American Society of Lubrication Engineers were announced at the fourteenth annual meeting in Buffalo, N. Y., last month.

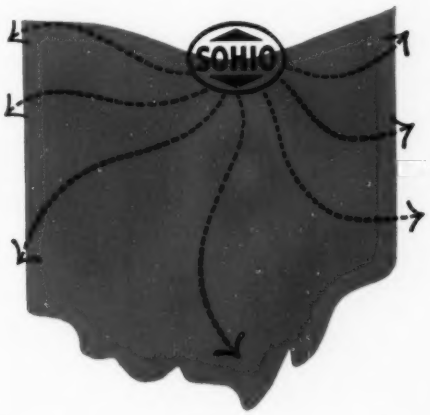
Dr. A. B. Wilder of E. I. duPont, Chicago, succeeds J. D. Lykins, Wheeling Steel, Wheeling, West Virginia as president. Mr. Lykins was named chairman of the presidential council, and L. O. Witzenburg, Farval, Cleveland, was elected vice president at large.

Other new officers include A. E. Cichelli, Bethlehem Steel, Bethlehem, Pa., secretary; C. C. Blaisdell, Penola Oil, Chicago, treasurer. C. L. Willey, Chicago, was reappointed as executive secretary.

Newly elected regional vice presidents include: D. P. Thomas, Columbia-Geneva Steel, western region; and J. Boase, Lincoln Engineering Co., Canada Ltd., Toronto, Ontario, Canada, Canadian region. Regional vice presidents retaining their office are: D. F. Wilcock, General Electric, Schenectady, eastern region; P. A. Asseff, Lubrizol, Cleveland, central region; and H. W. McCulloch, Jr., Shell Oil, Chicago, midwest region.

New additions to the board of directors for a three year term are: Dr. Earl G. Jackson, General Electric, West Lynn, Mass.; Howard T. Peeples, Timken Roller Bearing, Canton, Ohio. Dr. L. B. Sargent, Aluminum Co. of America, New Kensington, Pa. was elected as industrial director for a two year term.

Directors continuing their office include: R. J. Torrens, Eastman Kodak, Rochester, N. Y.; E. E. Bisson, NASA, Cleveland; B. T. Harding, Midwest Oil, Minneapolis; A. B. Two, Motor Oils Refining, Chicago, and M. M. Gurgo, Standard Oil of New Jersey, Pittsburgh.



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Industry News

USS Employs Use of Traveling Classroom

United States Steel has put a traveling classroom into service to give its employees theoretical knowledge and practical job know-how in the proper maintenance of plant lubricating equipment.

The classroom is a 35-foot mobile training unit, equipped with all the components of a centralized lubricating system.

Built by U. S. Steel's National Tube division, which has the responsibility for lubrication research for the corporation, the unit is mounted on a bus chassis so that it can be driven to any plant location.

The especially constructed steel body seats as many as fifteen people for demonstrations. Along both walls are removable panels upon which are mounted operating lubricating system components. These can be plugged into a built-in lubrication reservoir for "live" demonstrations with actual lubricating materials.

Work benches are located at each end of the demonstration area.

National Tube fitted the mobile unit with every type of lubricating equipment used in U. S. Steel plants. Employees at any plant thus can be given instruction on equipment identical to that in operation at their particular plant location. Demonstrators can convert from one type of equipment to another simply by placing different panels in the wall mounts.

The mobile unit carries its own generator and can operate independent of outside power. However, it can be plugged into a plant power source where this can be done conveniently.

It is completely air-conditioned and has a heating system for use during inclement weather. It also is equipped with a public address system, and carries a movie projector which is used in conjunction with a built-in screen.

Second Edition of Series Of Brochures

The second edition of a series of informative brochures on the care and use of stopper heads, prepared by the Joseph Dixon Crucible company, Jersey City, N. J., and offered to industry as a public service, is now in distribution. Copies in whatever quantities are desired may be had, at no cost, by writing to the company's crucible and refractories division.

The new edition deals with the assembling and drying of stopper rods. The inaugural brochure told how to eliminate stopper damage in plants and how to store them properly.

Handsomely printed in two colors on glossy stock, the new four-page brochure describes in text supported with photo illustrations and sketches, how to assemble stopper bolts, washers, keys, stopper plugs and sleeves.

For free copies, in any quantity desired, ask for No. 2 in the stopper head series, by writing to the Joseph Dixon Crucible company, Jersey City 3, N. J., crucible and refractories division.

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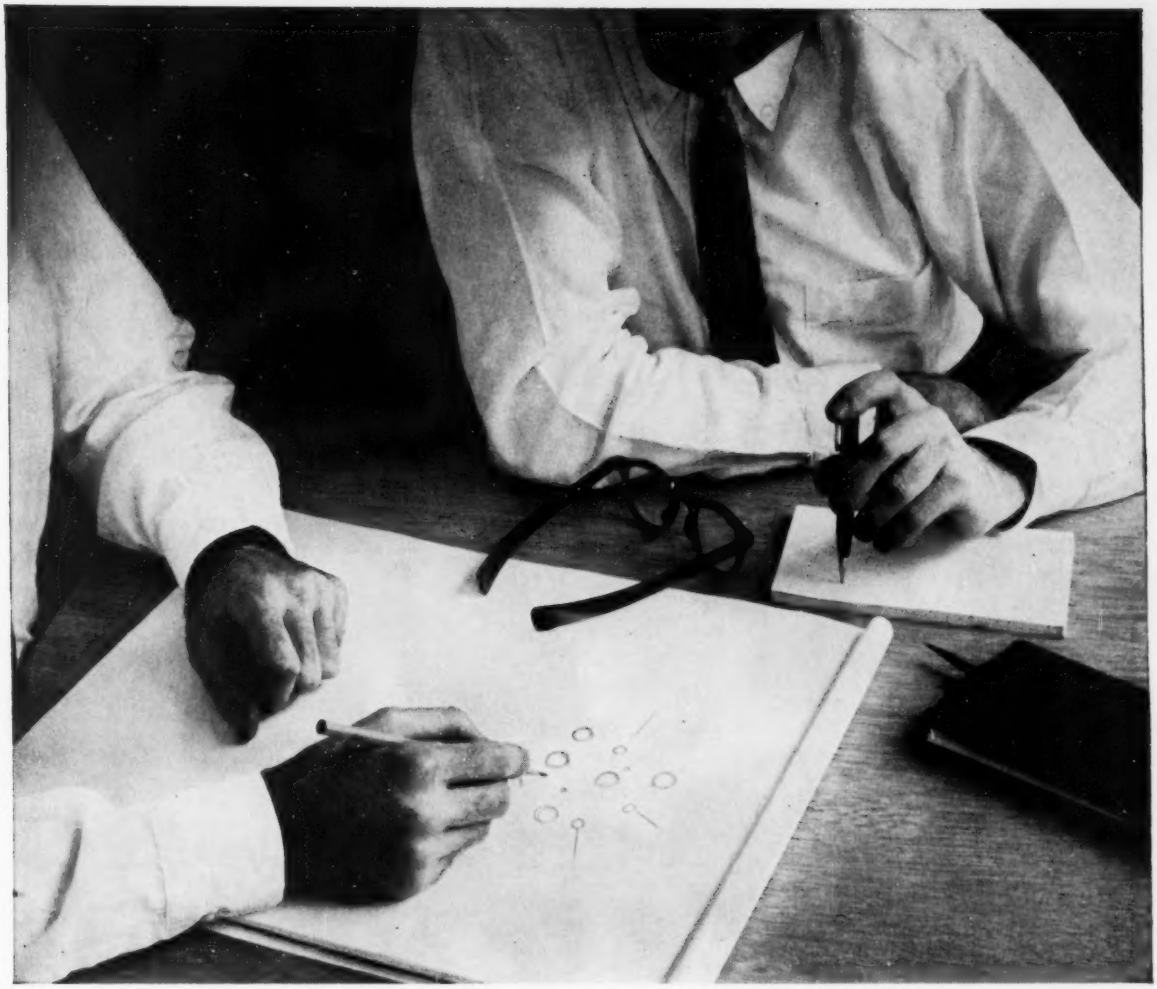

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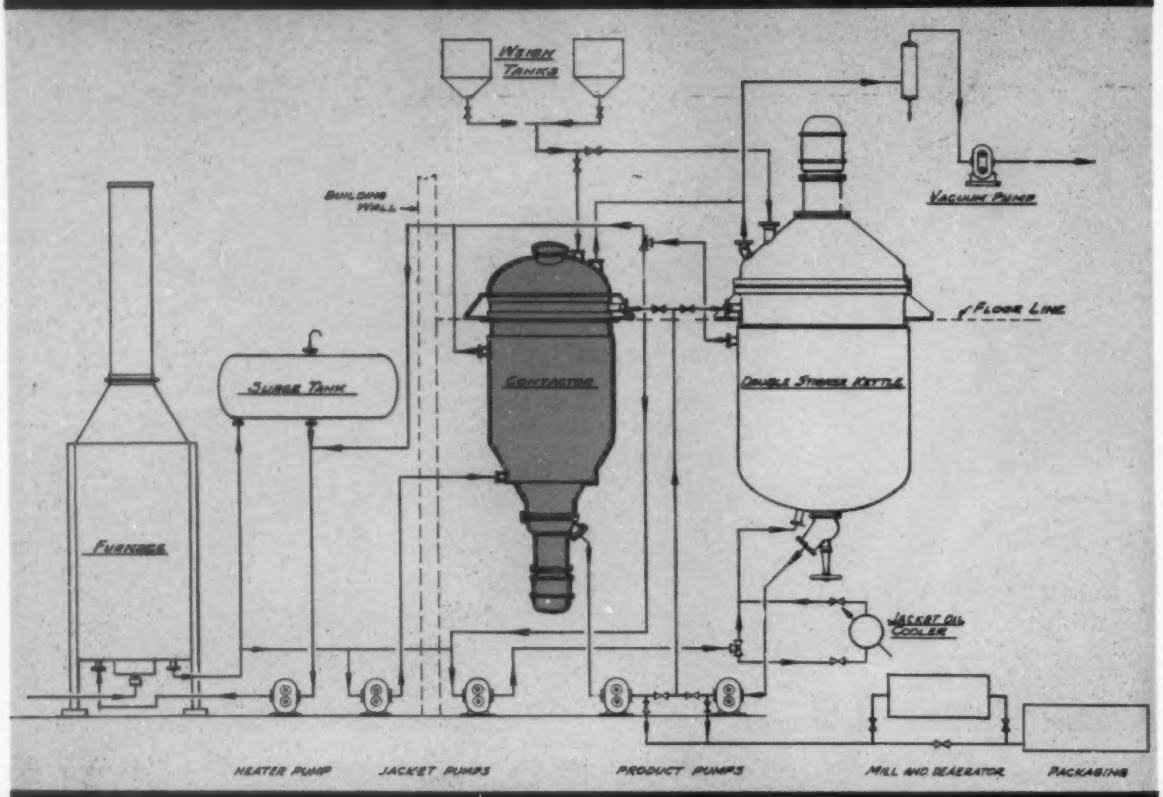

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